

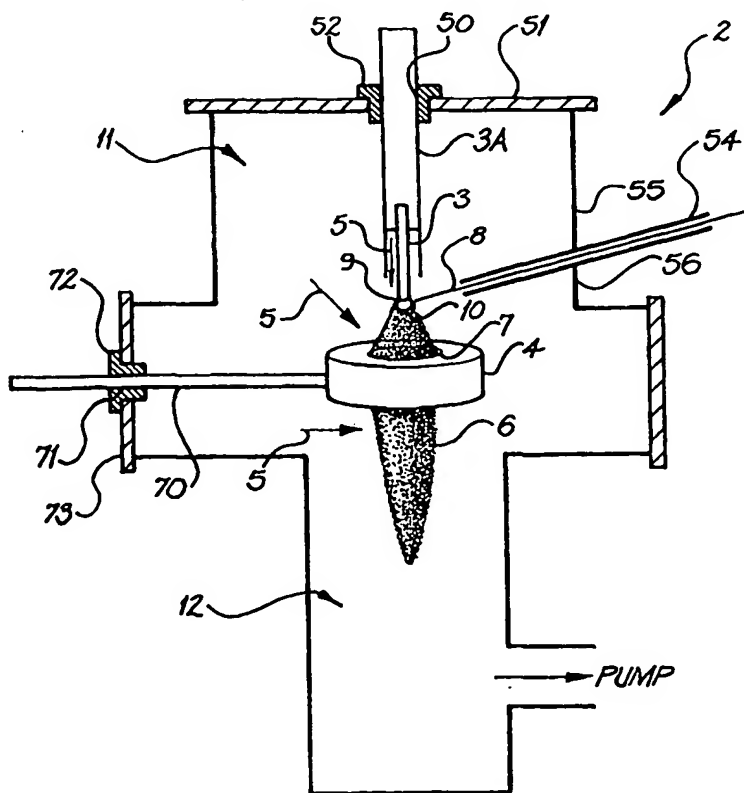
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(54) Title: METHOD AND APPARATUS FOR PRODUCING MATERIAL VAPOUR

(57) Abstract

An apparatus for producing material vapour for the production of powders or films comprises first (3) and second (4) electrodes mountable in a reaction vessel (2), and arranged to have a voltage applied to them to form an arc between them, the first electrode (3) being arranged to form at a part of the first electrode (9) a molten droplet or film (10) of a precursor material, when a precursor material (8) is moved towards the part, and wherein continued heating of the molten droplet or film causes vapourisation of material from the molten droplet or film to produce material vapour. The surface to volume ratio of the droplet or film (10) is selected in a manner such as to limit heat loss during an evaporation of material from the droplet or film, the heat loss being the portion of heat input into the surface of the droplet or film not used to evaporate material from the droplet or film.



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Method and Apparatus for Producing Material Vapour**Field of the Invention**

The present invention relates to the preparation of material vapour, particularly, but not exclusively, to the preparation of metal vapour which may subsequently be used in the synthesis of metal powders or metal compound powders or for depositing films of metal or metal-based compounds.

Background of the Invention

The production of chemically pure powder compounds and elements is desirable. Production of nano particles is desirable, for example, for the production of nano structured materials, which exhibit enhanced or unique properties.

The deposition of metal films or metal-based compound films also has many applications, for example in the aerospace industry and the electronics industry.

In order to produce metal powders and films, or metal compound powders and films, it is necessary to provide a source of metal vapour. There are a number of processes which are presently used to produce metal vapour for production of powders or films. All these processes evaporate metal from precursor metal material using one form or another of heating in a reaction vessel. Reaction conditions, such as temperature and pressure, are varied to produce either powders or deposit layers on a substrate.

With present processes, yields of material evaporation from the precursor metal are low and rates of production of powders and deposition of layers are also, therefore, low. The processes are also very energy-inefficient.

There are two types of low pressure arc deposition systems for deposition of thin films; cathodic evaporation systems and anodic evaporation systems. In a cathodic evaporation system, materials are evaporated from the cathode of negative polarity, and then deposited directly onto a substrate. In an anodic evaporation system, materials are evaporated from the anode, at positive

polarity, and the effects of convection and diffusion help deposit the metal vapour onto the substrate. In both processes, the substrate is usually shielded from the cathode to avoid ejection onto the substrate of
5 macro-particles from the cathode. A problem with both these processes is that the precursor metal is heated very inefficiently (due to heat losses associated with the relatively large volume of the heated materials and the bulk heating of these materials), so that the yield of
10 material evaporation and deposition on the substrate is low.

One particular process utilises an arc produced between a first electrode and a second electrode, the second electrode being in the form of a crucible containing
15 the metal precursor material. The crucible is heated by the arc to vaporise the metal precursor material (note that the crucible has a higher melting temperature than the melting temperature of the precursor metal material and will not melt at the temperature produced by the arc). A
20 substrate is mounted above the crucible. Metal vapour from the precursor material is deposited onto the substrate to form a film. Reactant gases may be introduced into the reaction chamber to react with the metal vapour in order to deposit a desired metal compound onto the substrate. In
25 this process, heating of the metal precursor material is very inefficient. Much of the heat may be conducted away via the crucible and by radiation. Further, all of the precursor material contained in the crucible has to be heated in order to produce the metal vapour, which adds to
30 the inefficiencies. Another problem is that metal vapour is emitted in all directions from the crucible. Only a certain amount is therefore deposited on the substrate, so that the amount of metal vapour deposited on the substrate is small compared to the total volume of metal evaporated.
35 Yet a further problem with this process, is that once the metal precursor material has been exhausted, the process

must be halted so that further metal precursor material can be added to the crucible.

In other techniques using low pressure arcs, the precursor metals are introduced in the gas phase through compounds containing the required metals. These techniques do not require vaporisation of solid materials.

Introducing the precursor metals in the gas phase through compounds containing them is, however, complex and limits the products that can be produced and also can introduce impurities from the elements contained in the gas phase compounds. Moreover, in this process the precursor materials may be very toxic.

The existing techniques using electric arcs for production of powders are based on evaporation of precursor material in a controlled pressure vessel. A similar electrode/crucible arrangement as discussed above may be used. The precursor metal is heated in the crucible to evaporate the metal and conditions are chosen so that powder is produced rather than a material layer. Usually, very low pressure conditions are used for depositing films and higher pressures, above 1 mbar and up to several atmospheres are used to produce powders. The rate of production of powders is very low due to the problems discussed above, in particular low evaporation rate from a crucible containing the precursor metal. Pulsed high currents in a wire have also been used for production of nano-sized particles.

High current arcs have also been used for powder synthesis. In one arrangement, (disclosed in US patent 5723027) an arc is used to create a plasma plume in a reaction vessel. Metal powder is injected into the plasma plume to form the required powder compound. A major problem with this technique is that the metal powder particles may not melt and evaporate before at least part of the particle has reacted to form the metal compound.

The result is powder particles which include a "core" of metal impurities.

In US 5460701, an arc is generated between a non consumable electrode and a consumable metal rod of the precursor metal, which operates as the other electrode. In order to prevent the entire precursor metal rod melting at once, it is held in contact with a water cooled copper anode. This anode acts to remove heat from the metal rod so that only a portion of the metal rod is melted at any time. The cathode is of tungsten held at an angle relative to the anode to form a plasma plume. The arc is operated in inert gas and a gaseous reagent is injected into the plasma plume to form the required compound.

Again, this process is inefficient, because much of the heat of the arc is carried away by the water cooled copper anode. If this is not done, however, there is a risk that the entire precursor metal rod would melt, which would result in the production of an uncontrolled amount of metal vapour, and also may affect the continuity of the product process.

There are therefore problems with the present processes of producing material vapour for use in manufacturing powders or depositing films.

Summary of the Invention

The present invention provides a method of producing material vapour for the production of powders or films, comprising the steps of providing first and second electrodes, applying a voltage to form an arc between the first and second electrodes, forming a molten droplet or film of precursor material at a part of the first electrode by heating precursor material by the arc, continued heating causing vaporisation of material from the molten droplet or film to produce material vapour.

The vapour also preferably forms a plasma via which current is continually transferred from the first electrode

(through the material droplet or film) to the second electrode.

Preferably, precursor material is provided from a precursor material member and the method includes the step
5 of feeding the precursor material member towards the part of the first electrode.

The present invention preferably optimises the efficiency of the material evaporation process by minimising the amount of heated material. Upon contact
10 with the part of the first electrode and plasma, the solid precursor material preferably melts, causing the formation [at the part of the first electrode] of a thin layer or small molten droplet. As this film or droplet is heated, heat losses from the molten material are minimal due to the
15 combined effects of its relatively small volume and the fact that the precursor material member is not heated significantly as the molten layer forms at the first electrode part. The solid precursor material preferably does not carry current. The current is transferred from
20 the first electrode to the second electrode through the plasma. As a result there is no ohmic heating within the solid precursor material, and also because most of the heat transferred from the plasma to the electrode is carried by charged particles, heat transferred to the precursor
25 material is only due to conduction. This heat serves to increase the temperature of only a small part of the precursor material member proximate the part of the first electrode. As material from this small part of the precursor material member is deposited on the part of the
30 first electrode, heat gained by the precursor material member is transferred to the droplet or film at the part of the first electrode. Therefore, preferably there are substantially no heat losses through the precursor material member. The only heat losses are radiation from the first
35 electrode (very small), and conduction through the bulk of the first electrode. Preferably, the first electrode has a

relatively small cross-section and a relatively low thermal conductivity, so heat lost through conduction is also low.

The precursor material is preferably mostly heated through its surface by heat transfer from the plasma.

5 Evaporation is preferably faster than other thermal processes within a range of temperatures, so the surface temperature of the material increases, resulting in a high evaporation rate.

The precursor material member is preferably in a wire
10 or rod form and is fed continuously towards the part of the first electrode to continuously produce vapour for a powder formation or material deposition process. The first electrode is preferably a rod electrode and the part of the electrode where the material layer or droplet is formed is
15 preferably its tip. The rod electrode is preferably cylindrical, and has a higher melting temperature and a lower thermal conductivity than the precursor. The precursor material may be metal.

Reagent gasses may be introduced into the plasma to
20 form metal compounds with the metal vapour. To produce powders, the process may be operated at pressures between a few millibars and several atmospheres. To produce deposits on a substrate, the process is operated with the substrate at very low pressures.

25 A wide variety of reagent gases may be introduced, such as nitrogen, hydrocarbons, etc.

Other working gases may also be introduced, such as inert gases, including helium, argon, Krypton, xenon. These can be used to effect arc operation. For example
30 argon may be introduced to alter heat transfer to the first electrode and/or protect the first electrode.

Instead of the second electrode being passive, as with conventional techniques, it may be used to direct the metal vapour plasma issued from the first electrode. Gas flow
35 and pressure variation in the reaction vessel may also be used to direct metal vapour plasma.

The present invention further provides an apparatus for producing material vapour for the production of powders or films, comprising first and second electrodes mountable in a reaction vessel, and arranged to have a voltage
5 applied to them to form an arc between them, the first electrode being arranged to form at a part of the first electrode a molten droplet or film of a precursor material when a precursor material is moved towards the part, and wherein continued heating by the arc of the molten droplet
10 or film causes vaporisation of material from the molten droplet or film to produce material vapour.

Preferably the first electrode is a rod electrode or a bar electrode and the part of the electrode on which the film or droplet is formed is the tip of the first
15 electrode. Preferably, the first electrode is cylindrical.

The second electrode may be a ring electrode with a hole in the centre which may be used to direct the metal vapour plasma, through the hole into a sectioned off part of the reaction vessel. This sectioned off part of the
20 reaction vessel may contain (mount) a substrate for metal deposition (at very low pressures) or may be used to collect powders produced at higher pressures. Gaseous reagents may be introduced into the reaction vessel to produce metal compounds. Other working gasses may be
25 introduced for control of the process.

The apparatus may also include a nozzle means for introducing gases to direct flow of the metal vapour plasma.

Preferably, the precursor material is introduced as a
30 rod or wire and is fed onto the tip of the first rod electrode. Preferably, the reaction vessel includes means for enabling the moving wire or rod of the precursor metal to be introduced to the tip of the first electrode and continuously fed thereto.

35 In accordance with a further aspect of the present invention there is provided a method of producing material

vapour for the production of powders or films, comprising the step of forming a molten droplet or film of precursor material, wherein a surface to volume ratio of the droplet or film is selected in a manner such as to limit a heat loss during an evaporation of material from the droplet or film, the heat loss being the portion of a heat input into the surface of the droplet or film not used to evaporate material from the droplet or film.

The present invention provides a master producing material vapour for production of powders or films, comprising the step of applying a voltage between first and second electrodes to form an arc between them, and feeding member of solid precursor material towards the arc in order to vaporise the precursor material to produce the material vapour, a solid precursor member not conducting any electric current.

Brief Description of the Drawings

Features and advantages of the present invention will become apparent from the following description of embodiments thereof, by way of example only, with reference to the accompanying drawings, in which;

Figure 1 is a schematic diagram of an apparatus in accordance with a first embodiment of the present invention, for producing powder particles;

Figure 2 is a schematic diagram of an apparatus of a further embodiment of the present invention for producing powder particles;

Figure 3 is a schematic diagram of an apparatus of yet a further embodiment of the present invention for producing powder particles;

Figure 4 is a schematic diagram of an apparatus in accordance with an embodiment of the present invention for producing material films;

Figure 5 is a schematic diagram of an apparatus in accordance with a further embodiment of the present invention for producing deposited films, and

Figure 6 is a schematic diagram of an apparatus in accordance with yet a further embodiment of the present invention for producing deposited films on substrates.

Detailed Description of Preferred Embodiment

5 Referring to figure 1, an apparatus for the synthesis of powders, particularly metal powders or metal-compound powders, is illustrated schematically in cross-section. The apparatus comprises a reaction vessel 2.

The apparatus comprises a first electrode 3 and a
10 second electrode 4. An electrode holder 3A mounts the electrode 3, and the holder 3A is mounted in a port 50 through a wall 51 in the reaction vessel 2. The holder 3A is conductive, and is mounted by means of plug 52. Means (not shown) are provided for applying a voltage between the
15 first electrode 3 (via holder 3A) and second electrode 4 to form an arc therebetween. Port means are provided for introducing working gases (indicated by arrow reference numeral 5), e.g., inert gases may be introduced to effect operation of the process and/or direct the flow of metal
20 vapour produced by the process. One of the port means may be within the electrode holder 3A. In this embodiment a first electrode 3 is in the form of a cylindrical rod, and the second electrode 4 is in the form of a cylindrical ring having a hole 7 in its centre opposite the first electrode
25 3. The second electrode 4 is mounted by an arm 70 which extends through part 71 in plug 72 in side wall 73 of vessel 2. The arm 70 may be hollow so as to introduce cooling fluid to the second electrode 4. The first electrode is positive and the second electrode is negative.

30 A precursor metal material 8 is introduced into the reaction vessel 2, being in the form of a moving wire 8 of solid metal material. The moving wire 8 is introduced through a hollow tube 54 mounted within a port 55 in a side wall 56 of the reaction vessel 2.

35 In operation, the moving wire 8 is fed towards the tip 9 of the first electrode 3. Due to the heating effect of

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the plasma 6 (the plasma being formed from metal vapour in the arc), a molten film or droplet 10 is formed at the tip 9 of the first electrode. Under the heating effect of the plasma 6, this molten film or droplet 10 vaporises. The
5 moving wire precursor material 8 is continually fed to the tip of the first electrode 3 so that the film or droplet 10 is maintained and so that the metal vapour is produced at a constant rate. Heat losses from the molten metal film or droplet 10 are minimised due to the combined effects of its
10 small volume, the rod configuration of the first electrode 3 and the low thermal conductivity of the rod 3. The material of the first electrode 3 is chosen so that its thermal conduction is lower than that of the precursor material 8 but its melting point is higher than that of the
15 precursor material 8.

The metal film or droplet 10, having a relatively large surface to volume ratio, is mostly heated through its surface through heat transfer from the plasma. Because
evaporation is faster than other thermal processes within a
20 range of temperatures, the portion of the heat input into the surface of the film or droplet 10 not used to evaporate material from the film or droplet 10 is limited. As
discussed above, metal material 8 is continuously fed to the electrode tip to compensate for the evaporated
25 materials.

The vessel is substantially divided into two sections by the second electrode 4 and the hole 7 in the second electrode 4 forms a passageway joining the two sections 11 and 12. Section 11 contains (mounts) the electrode and arc
30 and section 12 is used for collecting a resultant metal powder or metal compound powder (where a gaseous reagent is introduced) in the collection chamber formed by the section 12 of the vessel 2. A pressure differential may be
maintained between the two sections to drive the plasma 6
35 into the collection chamber 12. Flow of an inert gas 5 can also be used to assist directing the plasma 6 into the

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collection chamber 12. A pump (not shown) may be used to maintain low pressure in the vessel 2.

Figure 2 illustrates a further embodiment. Only part of the reaction vessel is shown in figure 2, for the purposes of clarity. The same reference numerals as used in figure 1 have been used in figure 2 for similar components, and no further description will be given of these components. The electrode holder 3A and the rest of the reaction vessel 2 is not shown. Further, the mounting of the second electrode 4 is not shown. Deposited powder 15 is illustrated, deposited on the inside of the walls of the reaction vessel 2. The gas introduced into the reaction vessel 2 may be inert gas for formation of the pure metal powder and reagent gas 13 for the formation of the desired metal compound. In this embodiment, the difference is that the plasma flow containing the evaporated materials is directed using a cathode jet, as illustrated. In this embodiment the first electrode 3 is an anode and the second electrode 4 is a cathode (in figure 1 embodiment, the electrode polarity may be the same as this or reversed).

Figure 3 schematically illustrates an embodiment utilising a different electrode arrangement. The first electrode 3 is still in the form of a rod. In this case, however the second electrode is in the form of a rod 20 as well. Gas is introduced as indicated by arrow 21 lengthwise into the collection chamber 22 which is an undivided collection chamber. The powder 23 is deposited on the walls of the collection chamber 22, as shown.

The pressure utilised in the reaction vessels is in the order of atmospheric pressure or just lower, in order to ensure that powder is produced.

The arrangements described above can be used for the production of powders of nitrides such as AlN and SiN, carbides such as WC, oxides such as AlO₃ and ZrO₂ and ultra-fine pure metal powder such as Al, W and Zr. Process

conditions can be altered to change the size of particles through control of the pressure and the gas flow, so different sized particles may be produced.

Observations have been made using the above
5 arrangements for several materials, including aluminium, tungsten, molybdenum, titanium and vanadium at different pressures. The applicants have found that the rate of evaporation is at least one order of magnitude higher than the conventional arrangements when a crucible or a metal
10 block is used. Also, the applicants have investigated synthesis of AlN at atmospheric pressure and have found that the processes is highly efficient for the production of AlN powders.

An embodiment of apparatus in accordance with the
15 invention for depositing metal films or metal-compound films on a substrate is illustrated in figure 4. The arrangement is essentially the same as the arrangement illustrated in figure 1 apart from the provision of the substrate 30 in the reaction vessel 2. Note that in figure
20 4 the components are illustrated schematically in the reaction vessel. The first electrode 3 holder and second electrode 3 holder are not shown. The operation of the arrangement is the same as the operation of the arrangement in figure 1, apart from that the reaction conditions are
25 altered by lowering the pressure so that metal film is deposited (or metal-compound film if a gas reagent is introduced) on the substrate 30. The same reference numerals are used for components which are similar to the components in figure 1 and no further explanation will be
30 given here with regard to their operation. Gases may be introduced in the same way as they are introduced in the arrangement shown in figure 1.

Note that for low pressure arcs, the high evaporation rate from the materials at the tip of the first electrode 3
35 helps minimise the formation of spots at the second electrode 4, resulting in diffuse arc-electrode attachment

when the second electrode is a cathode. This helps minimise ejection of macro-particles from the cathode, presenting a further advantage over existing techniques.

Figure 5 shows an embodiment for metal deposition or metal-compound deposition on a substrate 30 using a similar arrangement to that shown in figure 3, at lower pressure.

Figure 6 shows an embodiment for metal deposition or metal compound deposition on a substrate. Similar reference numerals are used for similar components to those of figure 1 and no further description will be given of those components. In this embodiment, the substrate 60 is placed to one side of the plasma 6 in portion 61 of the reaction vessel 2. A shield 62 is provided to shield the substrate from the second electrode 63 to prevent particles or metal vapour emanating from the second electrode from reaching the substrate 60. The second electrode 63 is a plate electrode (without a hole in the centre) mounted on mounting 64 which extends through port 65 in plug 66 in bottom wall 67 of the vessel 2. A pump (not shown) may be used to maintain low pressure in the chamber 2.

Any metal or metal-compound can be deposited on a substrate utilising this method and apparatus.

In all the arrangements illustrated in the drawings and discussed above, several metal material precursor wires could be fed onto the tip of the first electrode for deposition of multi-component films or preparation of multi-component powders. Alternatively, several systems as disclosed above could be used in one reaction vessel for production of multi-component powders or multi-component films.

Although the above description discloses the formation of metal vapours to form metal films or powders or metal-compound films or powders, it will be appreciated that other materials may be vaporised and used to form films or powders.

In the above example embodiment, the first electrode is a rod shaped electrode and the metal film or droplet is formed on the tip of the electrode. The electrode does not have to be a cylindrical rod. It could be a rectilinear bar and it could have non-uniform dimensions. The electrode could also be hollow and the precursor material could be introduced through a hole in the electrode (e.g., the electrode may be a hollow rod and precursor material may be introduced through a hole in the tip of the rod). The part of the first electrode where the material film or droplet is formed may be shaped to hold the material or to minimise heat losses through conduction away from the precursor material.

In the following, a description of the production of various example powders and example deposition of aluminium films will be given.

Example 1

Production of Aluminium Nitride (AlN) Powder:

For the production of AlN, the precursor materials are pure aluminium introduced in a wire form and the working gas is nitrogen injected above and below the ring (gas 13). The configuration is that presented in Figure 2. The first electrode (3) is positive (anode) and is of tungsten. The second electrode (4) is negative (cathode). This cathode is of water cooled copper and has a ring shape. The aluminium wire (8) is fed through entry (14) as shown in Figure 2, onto the tip of the first electrode (3). Heat from the arc causes the formation of molten metal droplet (10) of aluminium and the arc is struck between (10) and (4). For this particular example the pressure in the chamber is 1 atmosphere (but the pressure can be anywhere between 1 torr and several atmospheres). The wire feed rate depends on the arc current (normally higher for higher arc current).

Example 2

Production of Pure Aluminium Powder:

The configuration is that of Figure 1. The pressure is less than 10 torr. As in the previous example, the wire is of pure aluminium, but the injected gas (5) is pure argon (or any other inert gas). Here the gas is injected through (5). From a nozzle surrounding the first electrode. A further gas flow can be injected through (13) to cool the plasma down and stop further agglomeration of the powder.

A plasma plume forms as in Figure 1, and a black powder is collected in 12. This black powder is combustible upon exposure to air or oxygen. To prevent oxidation the powder has to be consolidated in an inert atmosphere.

Example 3**Production of Aluminium Oxide Powder:**

The same process in Example 2 but with oxygen is injected through (13) instead of argon. Also, the pressure can be between a few torrs and 1 atmosphere. The result is ultra-fine snow white powder of aluminium oxide.

Example 4**Production of Powder of Aluminium Carbide:**

The same as in Example 2, but a gas containing carbon (such as hydrocarbons) is injected through 5. The hydrocarbon gas dissociates in the plasma and the resulting carbon atoms react with the aluminium vapour issued from the tip of the first electrode to form aluminium carbide.

Example 5**Deposition of Aluminium Films:**

In this particular example, the arc is struck between the molten aluminium at the tip of the first electrode (as in Figure 1) and a water cooled cooper plate (instead of the ring electrode in Figure 1).

The pressure in the chamber is less than 100 mtorr. A substrate is placed inside the chamber (11). After a few minutes of arc operation a conducting film of aluminium of more than several micrometers thickness is formed on the substrate.

Example 6**Deposition of Aluminium and Nickel Films:**

In this particular example, the arc is struck between the molten metal which is of Aluminium or of Nickel at the tip of the first electrode and a water cooled copper plate 63 as in figure 6. The pressure in the chamber is less than 1m torr. A substrate 60 is placed inside the chamber. The substrate 60 is shielded from the second electrode 63 so that particles emanating from the second electrode do not reach the substrate and degrade the quality of the deposited film. After a few minutes of operation a conducting metal film of aluminium or of nickel depending upon the precursor metal is formed on the substrate. We have been able to obtain deposition rates of more than 100 micrometers per minute for aluminium and more than 10 micrometers per minute for nickel.

It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as shown in the specific embodiments without departing from the spirit or scope of the invention as broadly described. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of producing material vapour for the production of powders or films, comprising the steps of providing first and second electrodes, applying a voltage
5 to form an arc between the first and second electrodes, forming a molten droplet or film of precursor material at a part of the first electrode by heating precursor material by the arc, continued heating causing vaporisation of material from the molten droplet or film to produce
10 material vapour.
2. A method in accordance with claim 1, wherein the molten droplet or film of material has relatively small volume.
3. A method in accordance with claim 1 or claim 2,
15 wherein the precursor material is provided by a solid precursor material member positioned proximate to the first electrode part.
4. A method in accordance with claim 2, wherein the solid precursor material member is arranged so as not to
20 carry any current.
5. A method in accordance with claim 3 or claim 4, wherein the precursor material member is continuously fed towards the electrode part as the molten droplet or film is vaporised, whereby to produce a continuous source of
25 material vapour.
6. A method in accordance with claim 5, wherein the precursor material member is in a wire or rod form.
7. A method in accordance with any preceding claim, wherein the first electrode is a rod electrode and the part
30 of the electrode where the material layer or droplet is formed is the rod tip.
8. A method in accordance with claim 7, wherein the second electrode is a ring electrode having a hole in its centre through which the metal vapour may be directed.
- 35 9. A method in accordance with claim 7, wherein the second electrode is a plate electrode.

10. An apparatus for producing material vapour for the production of powders or films, comprising first and second electrodes mountable in a reaction vessel, and arranged to have a voltage applied to them to form an arc
5 between them the first electrode being arranged to form at a part of the first electrode a molten droplet or film of a precursor material when a precursor material is moved towards the part, and wherein continued heating of the molten droplet or film causes vaporisation of material from
10 the molten droplet or film to produce material vapour.

11. An apparatus in accordance with claim 10, wherein the reaction vessel incorporates a port for introduction of the precursor material in the form of a rod or wire.

12. An apparatus in accordance with claim 10 or claim
15 11, wherein the first electrode is in the form of a rod.

13. A method of producing material vapour for production of powders or films, comprising the step of forming a molten droplet or film of precursor material;
wherein a surface to volume ratio of the droplet
20 or film is selected in a manner such as to limit a heat loss during an evaporation of material from the droplet or film, the heat loss being the portion of a heat input into the surface of the droplet or film not used to evaporate material from the droplet or film.

25 14. A master producing material vapour for production of powders or films, comprising the step of applying a voltage between first and second electrodes to form an arc between them, and feeding member of solid precursor material towards the arc in order to vaporise the precursor
30 material to produce the material vapour, a solid precursor member not conducting any electric current.

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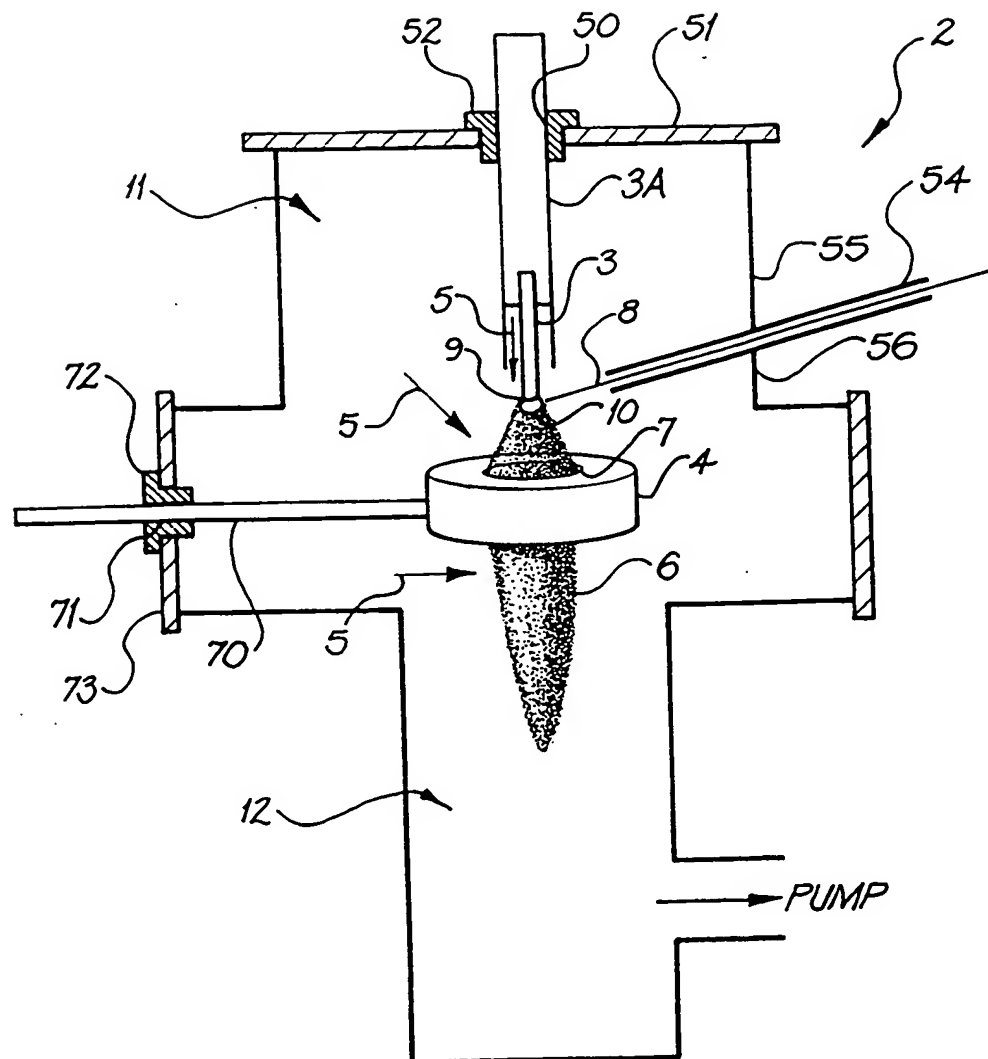


FIG. 1

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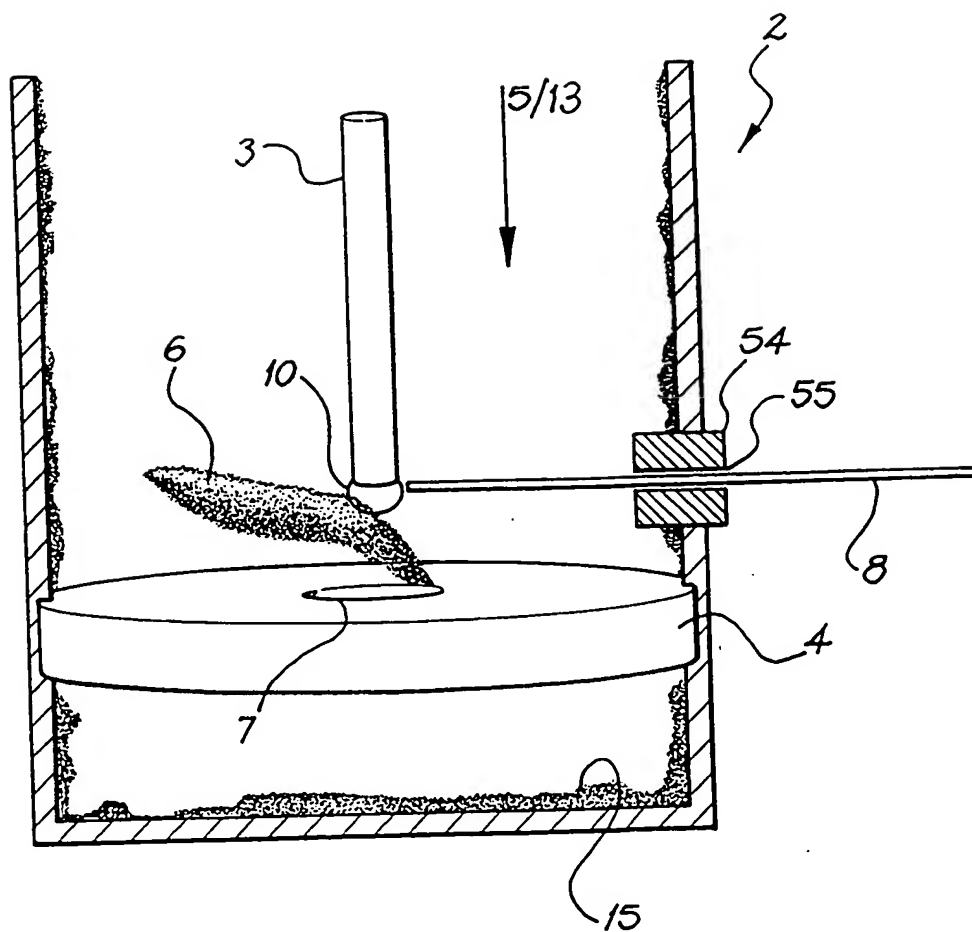


FIG. 2

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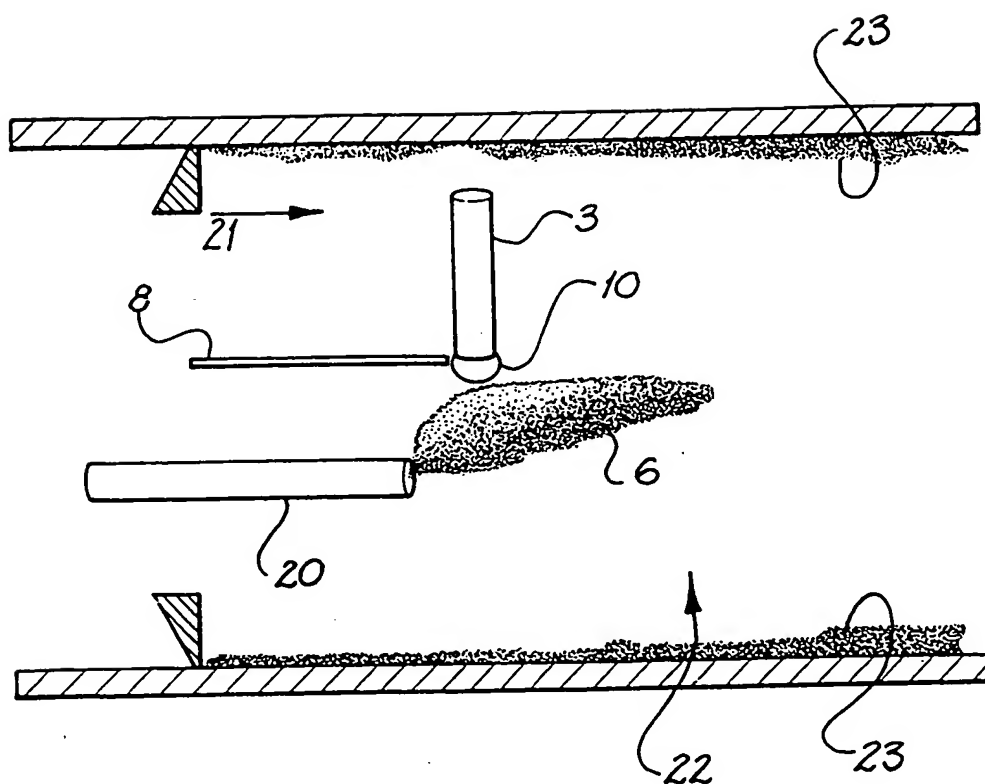


FIG. 3

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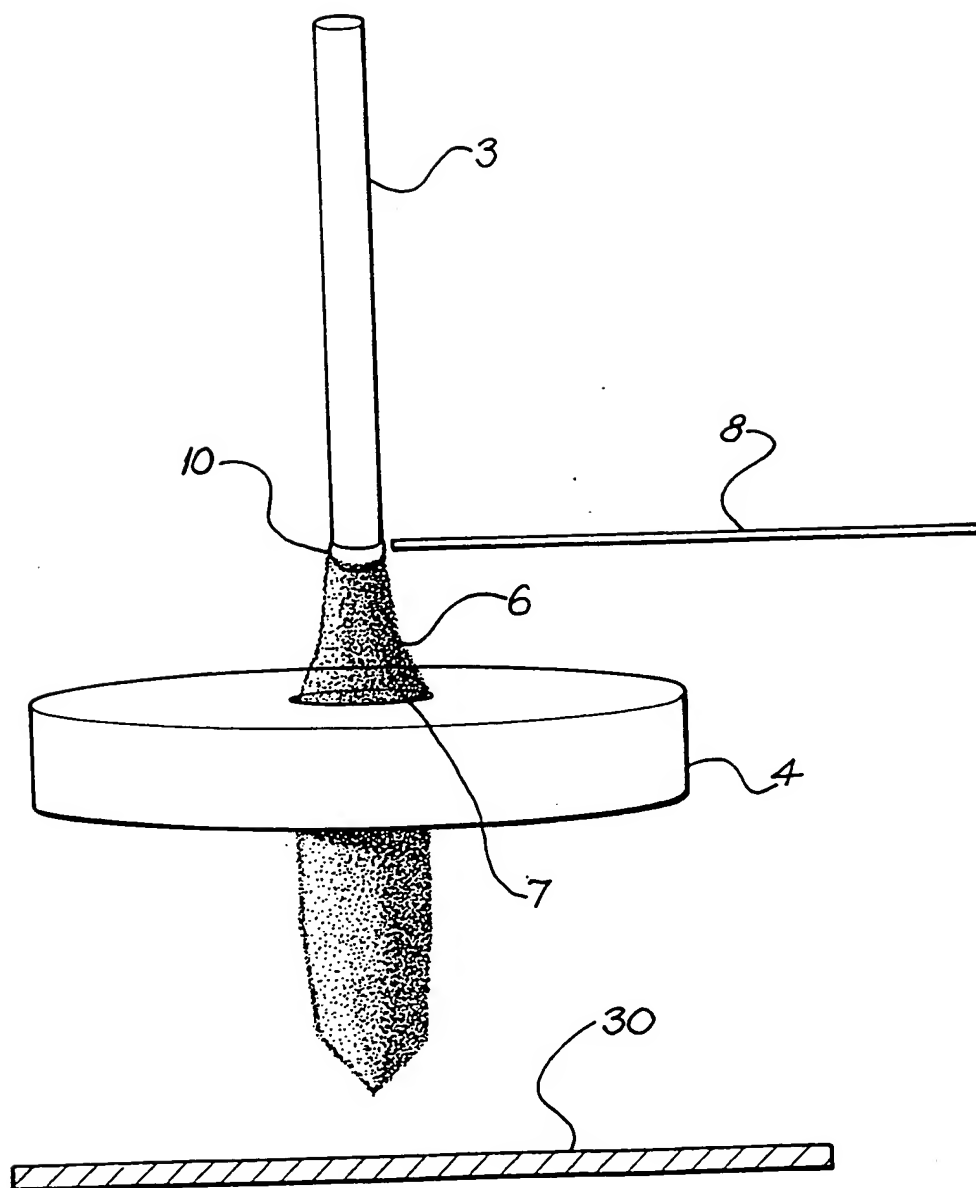
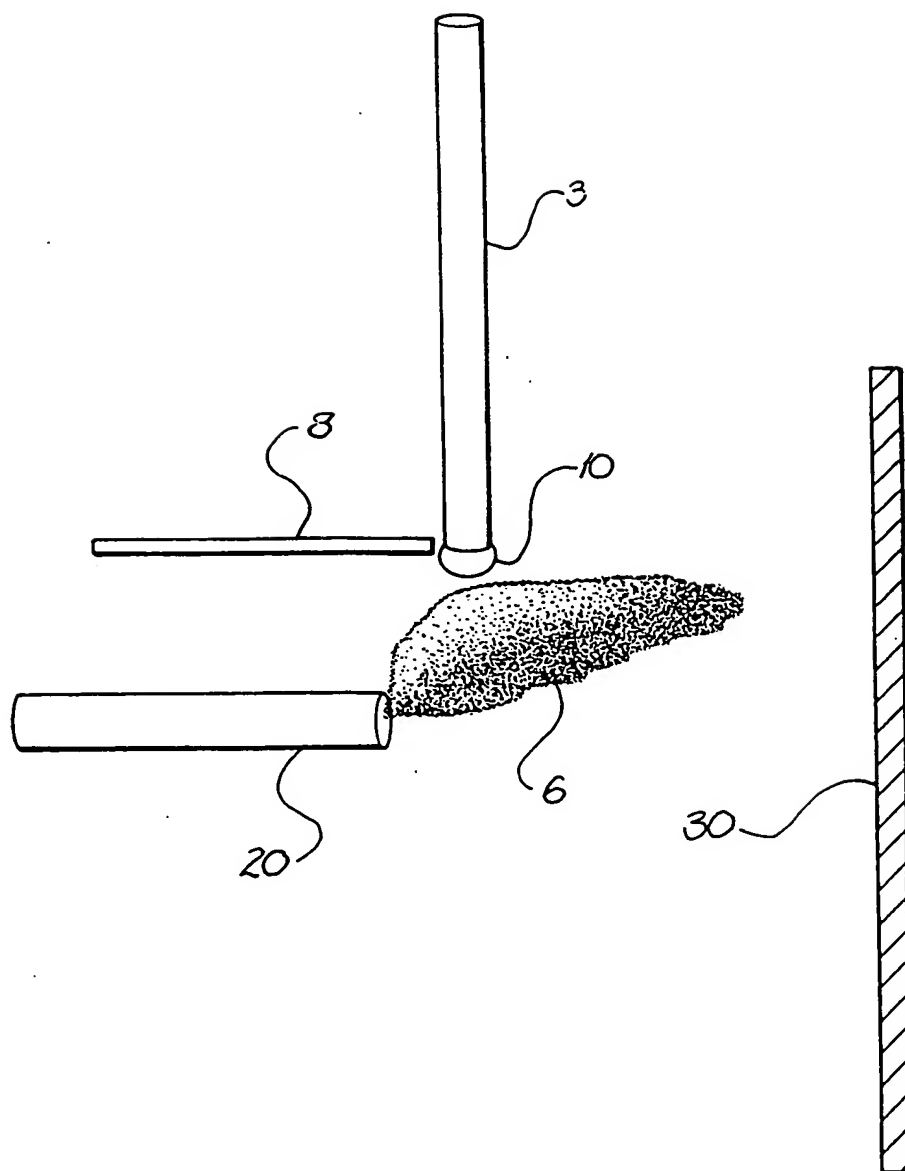


FIG. 4

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*FIG. 5*

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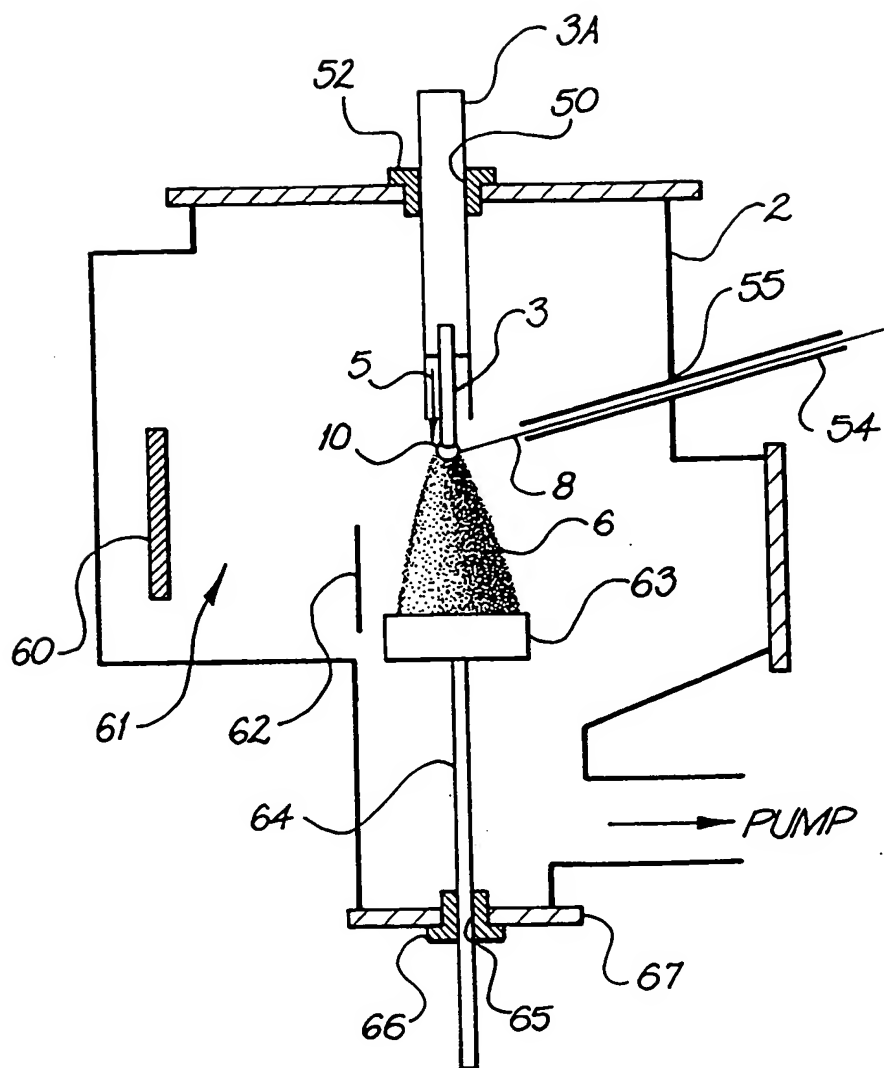


FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU 99/00588

A. CLASSIFICATION OF SUBJECT MATTER		
Int Cl ⁶ : B22F 9/16, C23C 14/24, 14/26		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) B22F 9/16, C23C 14/24, 14/26		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) Derwent On-line Abstracts, IPC as above, C23C-14/26 with keywords electr., anod. or cathod., period 1975-1999.		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E, A	EP 905272 A (UNITED TECHNOLOGIES CORPORATION) 31 March 1999 See abstract.	1-12
E, A	899356 A (RHEINMETALL INDUSTRIE AKTIENGESSELLSCHAFT (et al)) 3 March 1999. See abstract, also Derwent Abstracts Accession No. .99-144820/13, Class M13.	1-12
E, A	EP 885981 A (DR. EBERHARD MOLL GMBH) 23 December 1998 See abstract, also Derwent Abstracts Accession No. 99-037071/04, Class M13.	1-12
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"Z" document member of the same patent family</p>		
Date of the actual completion of the international search 8 October 1999		Date of mailing of the international search report 14 OCT 1999
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No.: (02) 6285 3929		Authorized officer G.J. BROXAM Telephone No.: (02) 6283

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 99/00588

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5723027 A (SEROLE) 3 March 1998. See abstract.	1-12
A	WO 96/16731 A (REY et al) 6 June 1996. See abstract.	1-12
A	DE 4405254 A (BENSTETTER) 24 August 1995. See abstract, also Derwent Abstracts. Accession No. 95-293705/39, Class L03.	1-12
A	US 5460701 A (PARKER et al) 24 October 1995. See abstract.	1-12
A	DE 4425221 C (VTD-VAKUUMTECHNIK DRESDEN GMBH) 24 August 1995 See Abstract, also Derwent Abstracts Accession No. 95-284422/38, Class L03.	1-12
A	EP 612859 A (VTD-VAKUUMTECHNIK DRESDEN GMBH) 31 August 1994. See abstract, also Derwent Abstracts Accession No. 94-219195/27, Class V05.	1-12
A	US 5238546 A (BERGMANN et al) 24 August 1993. See abstract, examples.	1-12
A	WO 93/02787 A (TETRONICS RESEARCH & DEVELOPMENT CO. LIMITED) 18 February 1993. See abstract.	1-12
A	DE 4221361 C (VTD-VAKUUMTECHNIK DRESDEN GMBH) 1 July 1993, See Abstract, also Derwent Abstracts Accession No. 93-206593/26, Class L02	1-12
A	DE 4117141 A (SEIDLER) 26 November 1992. See abstract, also Derwent Abstracts Accession No. 92-399947/49, Class P53.	1-12

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 99/00588

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.: 14
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
Claim 14 has been found to be unsearchable under Article 17(2)b because of defects under Article 17(2)b. Specifically, claim 14 is so unclear that no meaningful search could be carried out. It would appear that there is a missing phrase or line of text.
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

Claims 1-12 all relate to methods and apparatus in which material powders or vapours are generated by feeding a precursor material into the plasma arc formed between two electrodes.

Claim 13 relates to a method of controlling the surface/volume ratio of a droplet or film during evaporation in general. It is not limited to use in methods or apparatus according to any of claims 1-12.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: 1 - 12

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/AU 99/00588

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report			Patent Family Member			
EP	905272	NONE				
EP	899356	DE	19736028	IL	125579	
EP	885981	DE	19725930			
US	5723027	CN	1135190	EP	726806	FR 2724123
		WO	9607475			
WO	9616731	AU	43070/96	FR	2727635	
DE	4405254	NONE				
US	5460701	AU	73748/94	EP	711217	WO 9503907
		US	5514349	US	5874684	
DE	4425221	NONE				
EP	612859	NONE				
US	5238546	EP	444538	JP	6-041727	
WO	93/02787	NONR				
DE	4221361	NONE				
DE	4117141	NONE				
						END OF ANNEX